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Original Research

Making security glazing from modified TEMPO oxidized nanofibers and poly vinylbutyral

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Abstract

Safety glazing is a type of laminated glazing that holds together when shattered, due to the presence of an interlayer usually made of polyvinyl butyral (PVB). It is widely used in industrial application where the glazing could fall, or become a projectile to avoid serious injuries to humans after an impact with a foreign object. To replace the PVB, we have developed a modified cellulose nanofibers (NFC) by grafting poly(glycidyl methacrylate) with glycerol addition. This modification has improved the NFC pure performance as well as their compatibility with polar polymers. Therefore, this study reports the preparation and characterization of laminated glazing with composites of PVB reinforced with 40, 50 and 70% by weight of modified NFC. Composites interlayers are very transparent (up to 93% light transmission) with an interesting light character (up to 18.72% weight loss) in comparison to a PVB interlayer only. The chosen laminated glazing have been characterized by quasi-static (three-point bending) and dynamic impact loading (drop weight test). The quasi-static show the force–displacement curves of glazing while the dynamic testing give the energy absorption capability and maximum impact force. In our testing, the glazing with modified NFC have shown increased mechanical properties. Moreover, the laminated glazing made with an interlayer reinforced with 40% NFC exhibits the highest impact properties with a maximum force at break of 36,270 N and 24.81 J of absorbed energy. This study has shown that the modified NFC is indeed a lighter and environment-friendlier alternative for laminated glazing.

Keywords

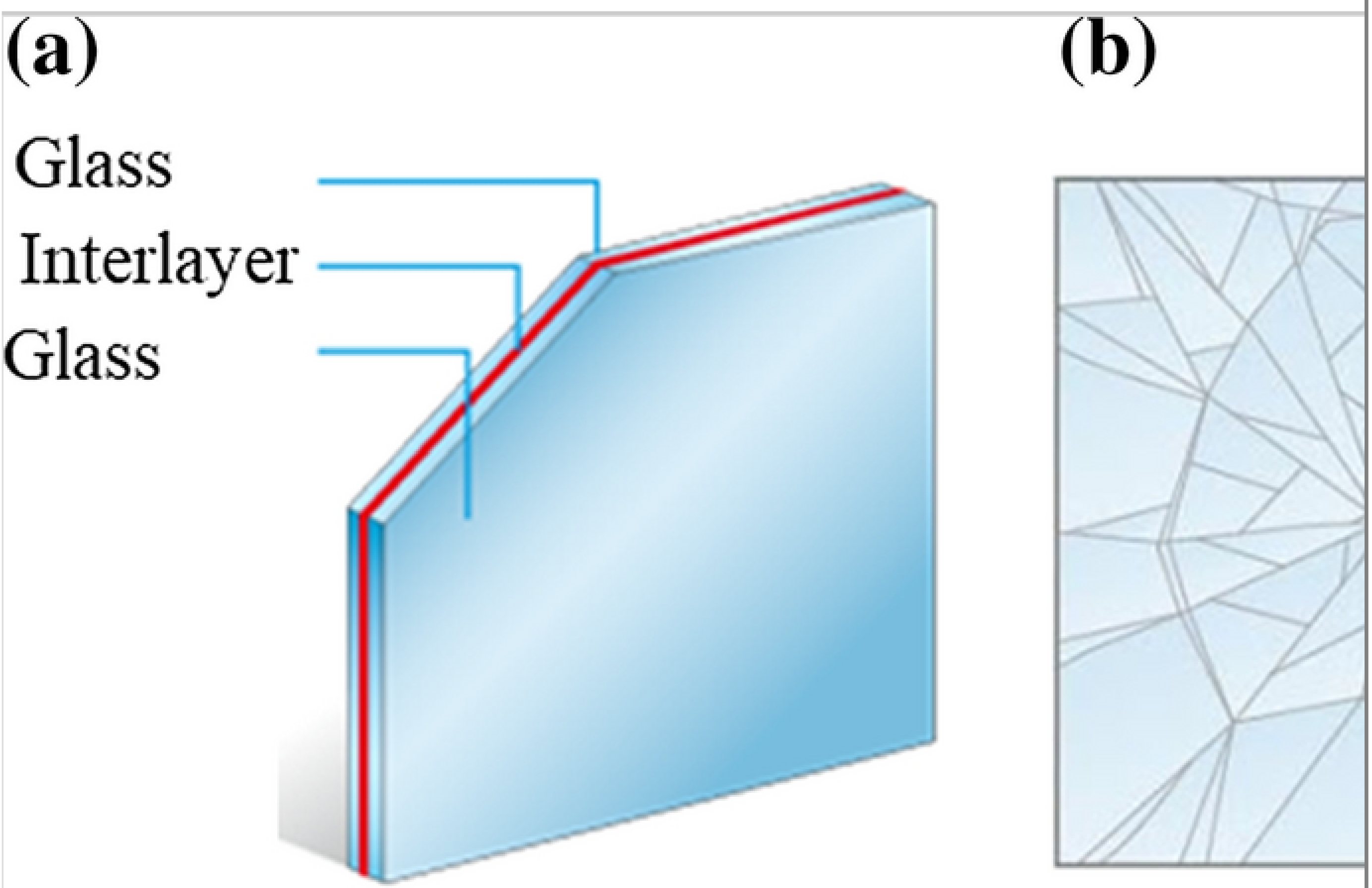
- Cellulose nanofibers (NFC)
- Glycidyl methacrylate grafting
- Glycerol
- Poly vinylbutyral
- Laminated glazing
- Security glazing

Introduction

In recent years, the total number of natural disasters, road accidents, and violent protests has globally increased. As a result, the demand for safety and bulletproof glazing is raising (Martín et al. 2020). Once usually used for car windshields (Gao et al. 2019) and special needs in structural elements of some buildings (Pariafsai 2016), such glazing is now also of interest for more common needs as glass facade windows of supermarkets or administrative buildings (authorities and services, precincts, etc.). Laminated glass is composed of at least two glass sheets, with a transparent plastic film in between, but many more are required for bulletproofing. In the fabrication process, the glass panels are being bonded under pressure to the interlayer into an autoclave thanks to the chemical link between silanol groups (glass) and hydroxyl groups (polymer) (Galuppi and Royer-Carfagni 2014). Thus in case of an event, the cracked fragments are kept glued to the interlayer, minimizing potential glass projections, maximizing the glazing resistance and security (Maury et al. 2019) (Fig. 1).

Fig. 1

Laminated glazing **a** normal, **b** broken



Several types of polymers are used in security and bulletproof glazing such as polyvinyl butyral (PVB), ethylene–vinyl acetate (EVA), ionomers, polycarbonate and thermoplastic polyurethane (TPU) (Martín et al. 2020). Usually, the choice of an interlayer differs from the requirement of each application. Nowadays, PVB is the most commonly used polymer in laminated safety glass especially as interlayer in car windshields and facades (Gao et al. 2019; Grethe et al. 2018; Huang et al. 2018). However, this polymeric material is a non-biodegradable, expensive, and petroleum-based thermoplastic (Cha et al. 1998). Thickness, hence weight of such laminated glazing is also a disadvantage, with the increasing demand for fuel economy and gas emission control improvement. Indeed, there is a great deal of interest in reducing any weight to improve the performance of aircraft and vehicle (Government of Canada. 2018; Immarigeon et al. 1995; Joost 2012). Taking cars as an example, 12–15% of the energy in the fuel is lost to move the car forward (Joost 2012).

The idea behind our study is to introduce cost-effective, renewable and wood-based interlayers with competitive mechanical properties in such glazing. Recently, we have successfully synthesized a composite of polyvinyl butyral reinforced with modified (Lassoued et al. 2020) and unmodified (Maury et al. 2019) TEMPO oxidized nanofibers (NFC). The synthesized composite with pure NFC (Maury et al. 2019) has shown increased mechanical characteristics, showing potential applications for safety and bulletproof glazing. However, while being satisfactory in light transmission, the optical quality had to be upgraded. This paper is in the continuity of our previous work, addressing this issue through finding a more efficient glazing process. Our solution is to chemically modify the cellulose nanofibers by in situ polymerization using glycidyl methacrylate as a monomer followed by the addition of a plasticizer, glycerol. This key step allows the synthesis of flexible and more hydrophobic NFCs films, thus increasing their compatibility with polar polymer matrices and the transparency of the resulting composites. Furthermore, the addition of modified cellulose nanofibres as reinforcing agents in the polymer composite provides an improvement in the absorbed energy and the elongation at break compared to an interlayer of PVB alone. In this article, laminated glazing samples made with different combinations of glass sheets, PVB and modified NFC mixes as interlayer are tested. The characterization of their behavior is carried out under static stresses, flexion tests, and dynamic stresses, drop weight testing.

Experimental

Materials

The 4-acetamido-2,2,6,6 tetramethylpiperidine-1-oxyl (TEMPO) oxidized cellulosic nanofibres, as a NFC gel at 3.3% in weight, is produced through selective oxidation with a TEMPO catalyzer and sonication treatments of a bleached Kraft pulp of resinous wood (Loranger et al. 2011; Paquin et al. 2013; Rattaz et al. 2011). The NFCs obtained has a carboxyl content of 1700 mmol/kg. PVB used is a copolymer of vinyl butyral-*co*-vinyl, alcohol-*co*-vinyl acetate with a molecular weight of 50,000 to 80,000 and was supplied by Sigma-Aldrich™ (Canada). Glycidyl methacrylate (GMA, 97%,) and glycerol (99%) were purchased from Sigma-Aldrich™ (Canada). Ethanol (Ethyl alcohol anhydrous) was obtained from Greenfield Global™ (Canada). Chemicals are used as received without further purification. Clear glasses intended for residential interior and exterior doors or windows (thickness of 6 mm) are used for the glazing. The glass sheets dimensions are (10 × 10 cm^2) for flexion testing (labelled as of type C) and 30 × 30 cm^2 for drop weight testing (labelled as of type D).

Preparation of the interlayers

The preparation of modified NFC films is carried out based on our previous work (Lassoued et al. 2020). Firstly, a transparent and aqueous dispersion of pure NFC is obtained after centrifuge NFC gel at 13,000 rpm for 15 min and reconcentration under vacuum evaporation (1% in weight dispersion). Subsequently, under a nitrogen atmosphere, 0.1728 g of ammonium persulfate (APS) is added into 7 g of NFC in water for 1 h. Then 16.15 g of Glycidyl methacrylate (GMA) is added at 40 °C. The mixture is continuously stirred for 48 h to complete the grafting reaction. Finally, 1.9 ml of glycerol, as a plasticizer, is added at 70 °C for 24 h.

PVB/NFC sandwich composites are synthesized by using the coating methodology reported in previous work of our group (Lassoued et al. 2020; Maury et al. 2019). In a mold, with a dimension of (20 × 20 cm^2) to prepare 4 glasses sheets of (10 × 10 cm^2) and (40 × 40 cm^2) to prepare 1 glass sheets of (30 × 30 cm^2), the modified NFC dispersion is poured onto an already dried PVB film synthesized with half of the total mass of PVB for the sample. After 48 h, the remaining amount of PVB is added, producing a sandwich-like composite. The composition of NFC/PVB interlayers 20 × 20 cm^2 and 40 × 40 cm^2 is illustrated in Table 1.

Table 1

The compositions of NFC/PVB interlayers 20 × 20 cm^2 and 40 × 40 cm^2

Samples	**PVB (g)**	**NFC (g)**	**Mass fraction of NFC (%)**
Interlayers 20 × 20 cm^2			
C1	20	0	0
C2	12	8	40

Samples	PVB (g)	NFC (g)	Mass fraction of NFC (%)
C3	10	10	50
C4	6	14	70
Interlayers 40 × 40 cm^2			
D1	40	0	0
D2	24	16	40
D3	20	20	50

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Laminated glazing samples fabrication

Glazing are assembled using the hot-pressing method as optimised by Maury et al. (2019). During the lamination process, the interlayers are placed between two sheets of glazing and then subjected to a pressure of 2 bars for 30 min at 110 °C. After pressing, the glazing are let to cool down at room temperature before any characterization or extensive manipulations.

Characterization

Optical properties

Light transmission of films and glazing is measured by a Tint Meter Inspector Model 200™, manufactured by Laser Labs, Inc.™ of Scituate, Massachusetts. It is commonly used by Canadian police forces to measure the total amount of Visual Light Transmission through a car window and any coatings (tint film) on that window. While only part of the optical properties, light transmission is neither less an important component of the glazing transparency. As the main goal of this study was to assess if the new synthesis path was not downgrading light transmission of our former research, while observing a better transparency (aka refractive property) of our synthesis, no other optical qualities are quantified.

Thickness

A Lhomargy™ micrometre is used for measuring the thickness of the test specimen to an incremental discrimination of 0.01 mm.

Scanning electron microscopy (SEM)

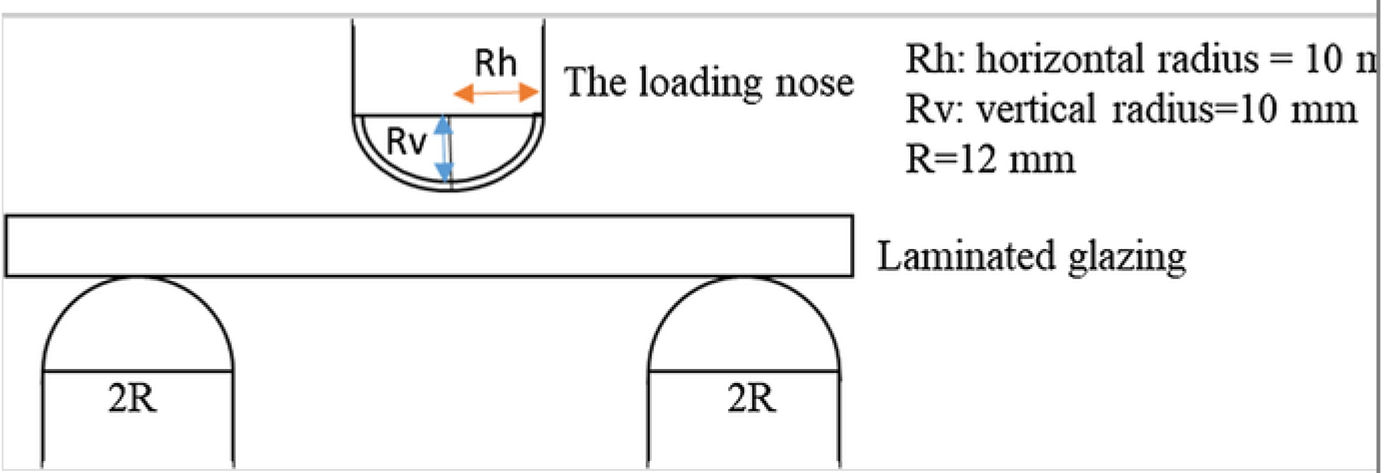
A JEOL JSM T300™ scanning electron microscopy is used for SEM imaging. An acceleration voltage of 5 kV and 2500 × magnification is performed to observe the morphology of the cross-section of the samples. All samples are deposited on a steel plate and coated with a mix of gold and platinum.

Three-point bending

The flexural strength of laminated glass samples is determined by three-point bending tests using universal testing machine (Instron 4201™) equipped with a 5000 N compression load cell. Dimensions and conditions for performing tests used are following the ASTM D790-03 specific to laminated glazing. The loading model is presented in Fig. 2. The specimens are placed on two supports then the loading nose is applied to the center of the beam until failure. Three glass plates for each type of interlayer were tested or measured once and the results presented are the means, min and max of each of these measurements.

Fig. 2

Loading model for three-point bending tests according to ASTM D790-03



Drop weight test

The impact tests are carried out using a CEAST 9350™-drop weight with a load cell of 45 KN. This test characterizes the impact resistance of materials under dynamic stress by measuring its impact force resistance and energy absorption capacity. By dropping a mass of 25.47 kg and an impactor diameter of 10 mm at a speed in the order of 4.21 m/s, the energy delivered by the latter on the glazing is 226 J. This value was determined as a target by Maury et al. (2019).

Results and discussion

Optical and structural properties

While not the main goal of this project, photographs of Fig. 3 (left) support that the proposed composites, as such as 50% NFC, have a similar transparency as NFC film alone, as shown by the clear readability of the various texts read through. It is, nevertheless acknowledged that a small and very light yellow tint is noticeable for all NFC films in comparison to the PVB only film, that would require further research to assess this chromatic shift, its operative repercussion, if not its correction. Table 2 shows that the interlayers obtained in our project present light transmission values (90–93%) similar to PVB (C1).

Fig. 3

Films photography (left) and cross-section micrographs (right) of PVB, modified NFC (Lassoued et al. 2020) and NFC/PVB interlayers (C3, 50% NFC)

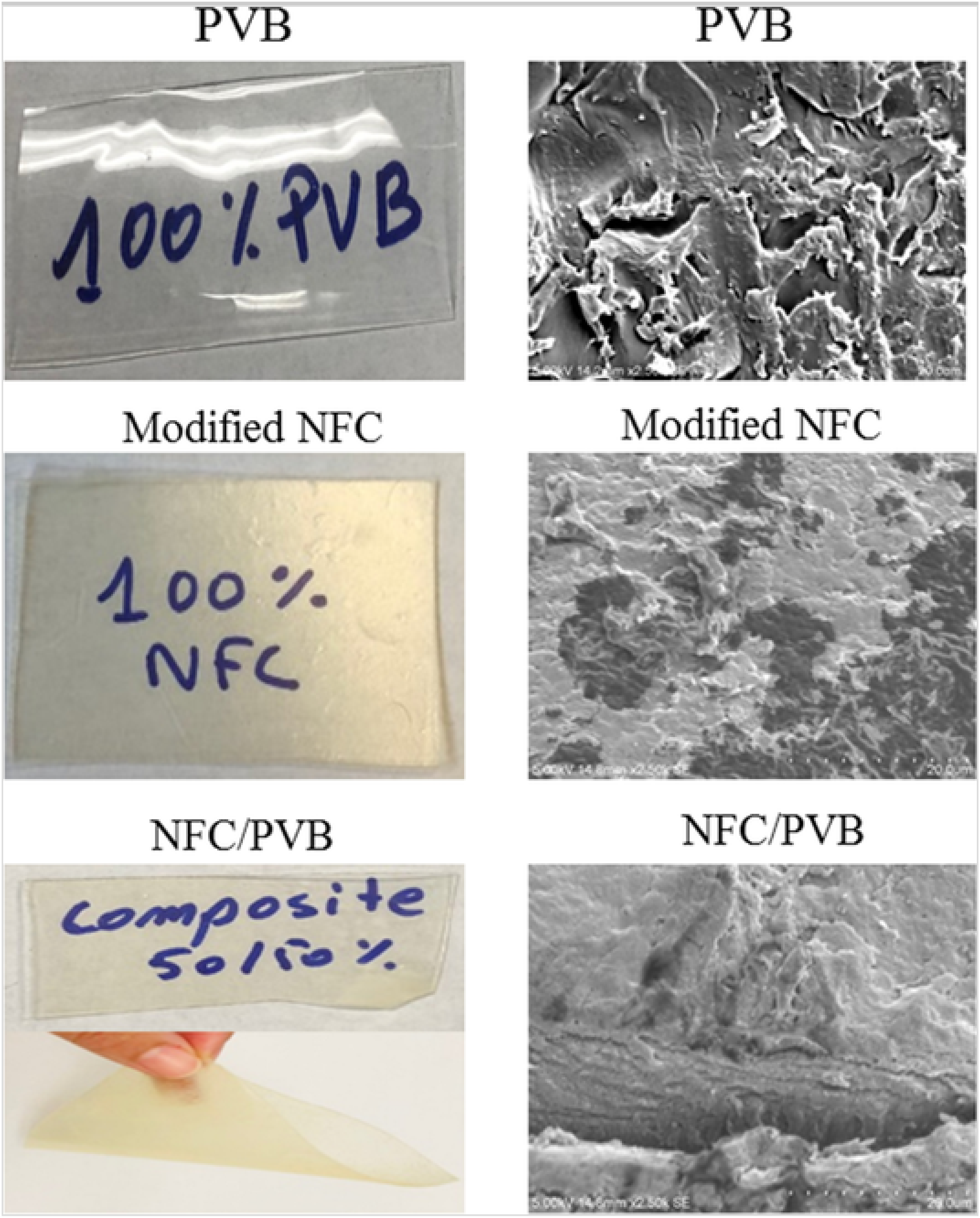


Table 2

Characteristic of films and laminated glazing (10 × 10 cm^2)

Samples (% NFC) n = 3[a]	Interlayers properties			Glazing properties			
	Surface density (kg/m^2)	Thickness (mm)	Light transmission (%)	Load (N)	Young's modulus E (MPa)	Extension (mm)	Ener at brea (J)

[a]Number of glass plates tested

Samples (% NFC) n = 3[a]	Interlayers properties			Glazing properties			
	Surface density (kg/m^2)	Thickness (mm)	Light transmission (%)	Load (N)	Young's modulus E (MPa)	Extension (mm)	Ener at brea (J)
C1 (0% NFC)	0.45 ± 0.02	0.45 ± 0.006	93	3430.5 ± 721.76	1.93 ± 0.35	3.57 ± 0.16	1.57 0.65
C2 (40% NFC)	0.41 ± 0.04	0.25 ± 0.006	93	3907.00 ± 618.00	2.01 ± 0.10	3.82 ± 0.10	1.82 0.20
C3 (50% NFC)	0.38 ± 0.04	0.30 ± 0.006	93	3385.00 ± 94.00	2.09 ± 0.15	3.93 ± 0.09	1.80 0.06
C4 (70% NFC)	0.36 ± 0.03	0.35 ± 0.006	90	2997.50 ± 118.00	1.80 ± 0.44	3.61 ± 0.11	1.31 0.06

[a]Number of glass plates tested

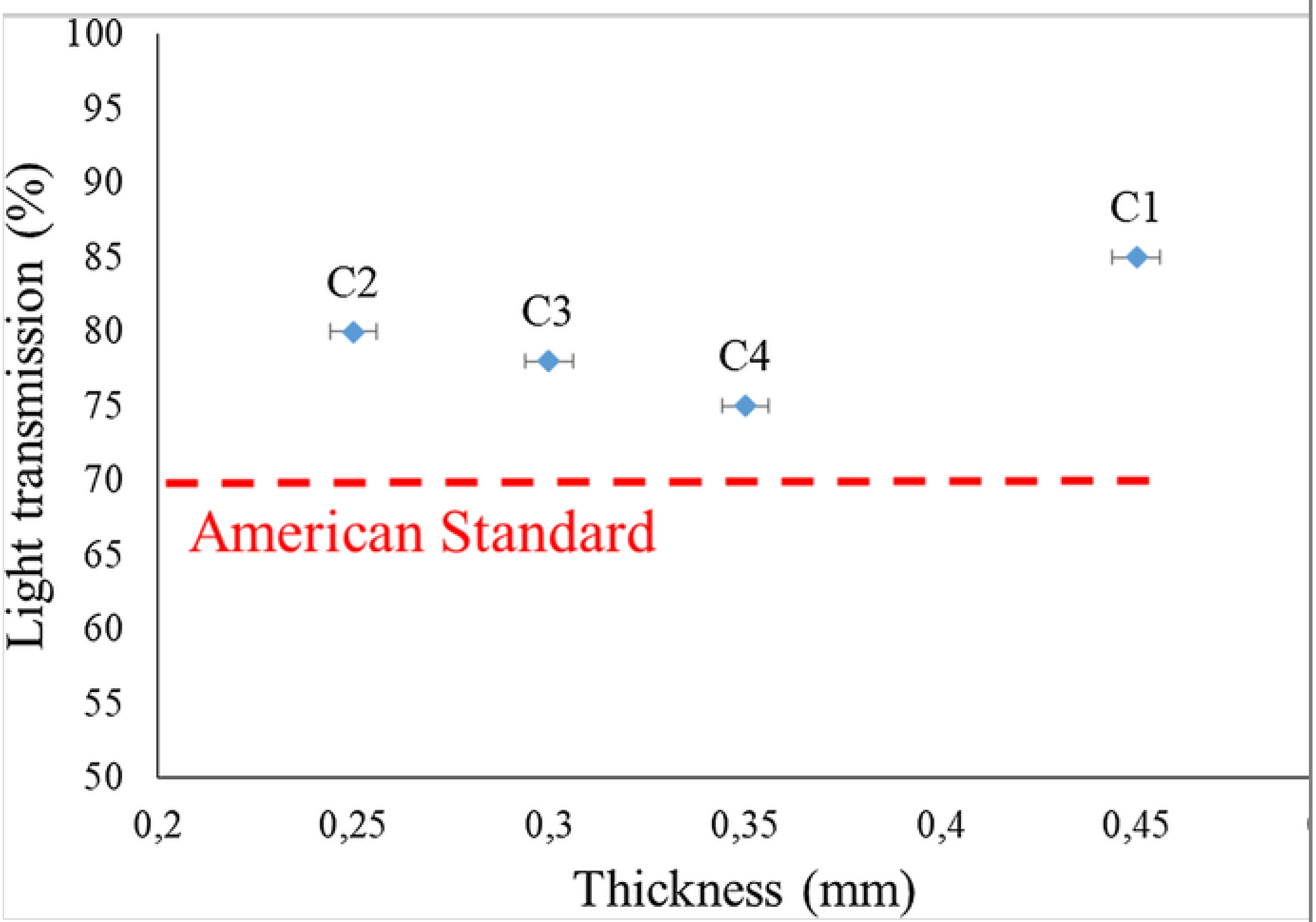
Even if the fabrication process involves layer-by-layer methodology (Lassoued et al. 2020; Maury et al. 2019), the allegedly synthesized sandwich interlayers are found to be a single pliable and stretchable layer. Figure 3 right shows the SEM micrographs of the cross-section of PVB, modified NFC and nanocomposite NFC/PVB. From this figure, it is virtually impossible to discern the layers. Indeed, the morphology of the multilayer composite appears as a single homogeneous layer. Contrary to being inert, the modified NFC seems to penetrate deeply the already dry PVB film found on the bottom. In the second part of the casting, the pouring of the PVB dispersion on the now dried modified NFC film seems to also exhibit the same behaviour. This phenomenon can be explained by the increased compatibility between the PVB polymer and the modified NFC, helping the diffusion of both solution into an already dried film. The absence of voids, fiber fractures, and a pullout in SEM micrographs seem to confirm our hypothesis and could be reported as an effective interfacial adhesion in biocomposites as described by Khan et al. (2018). Further, these observations are also accountable in the thickness reduction of the composite, each time a fraction of the PVB is replaced by the modified NFC.

Light transmission of the latter is also measured 3 times, showing no variation (Table 2). Interestingly, Fig. 4 presents the light transmission of the glazing as a function of the thickness of the interlayers. First of all, the achieved results are very promising and fulfill the requirements of the American Standard Safety Code about the minimum light transmission of automobile windshields (70%) (LaMotte et al.

2000). Secondly, the best light transmission value is still obtained with a PVB interlayer alone (85%). Nevertheless, the light transmission of composites reinforced with 40 and 50% NFC remains relatively close to that of PVB, at 80 and 78% respectively. The slight decrease, in comparison the intercalary alone, in general light transmission of composites is most likely attributed to our hot-pressing methodology. It is well known that heat further increase the yellowing of NFC interlayers (Enayati et al. 2016) by the modification of chromophore groups on the residual lignin but (Enayati et al. 2016) and might also affect negatively other components of the composite. In fact, according to Miraftab et al. (2015) yellowing indicates a modification of the chemical structure by forming conjugated double bonds due to dehydration phenomenon, which could occur on PVB, GMA, etc. Despite the yellowing from the film drying and hot-pressing, our result show that by limiting the thickness of the NFC composites, an improved optical quality of the glazing can be achieved.

Fig. 4

Light transmission as function of interlayers thickness for C1 (0% NFC), C2 (40% NFC), C3 (50% NFC) and C4 (70% NFC)

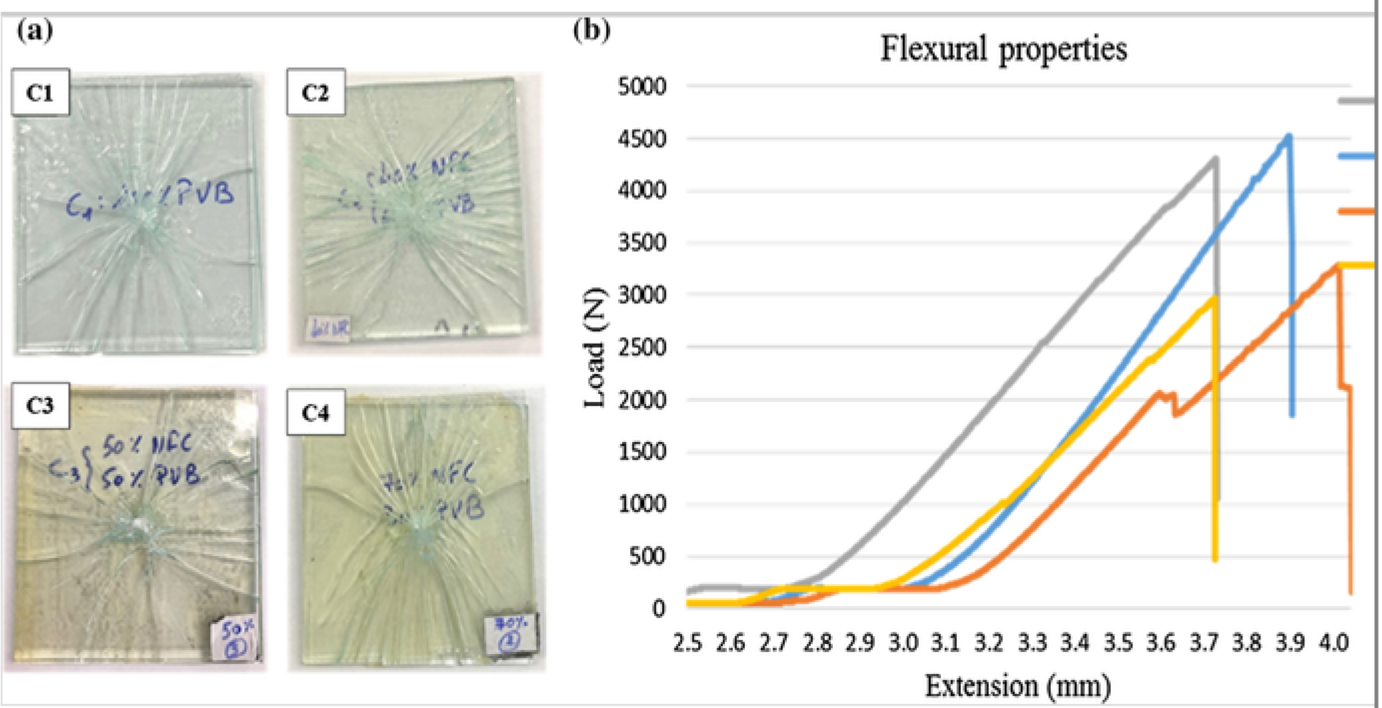


Flexural properties of laminated glazing

While Fig. 4 shows that better optical properties could be achieved if the thickness is minimized, the limiting factor of such reduction is, of course, the mechanical properties (Fig. 5). As for the optical properties, a qualitative appreciation can be achieved of the glazing. Figure 5a shows that after fragmentation of the glass, the interlayer does retain the glass pieces, thus avoiding potential fragments projection. This also gives the glazing residual rigidity, guaranteeing the stability of its implementation and ensuring the retention of the impact body.

Fig. 5

a Laminated glazing photographs after testing and **b** three-point bending load-extension curves



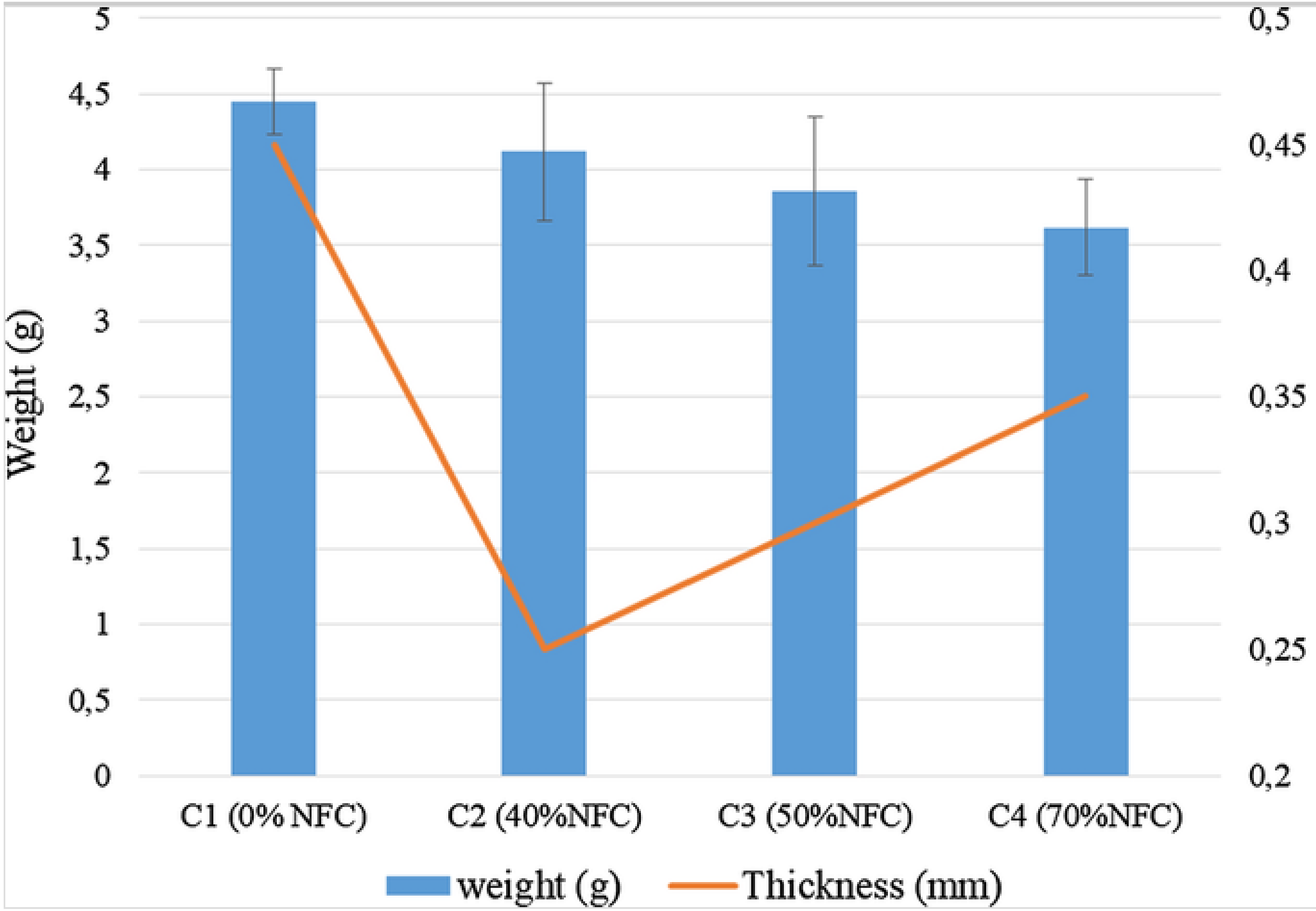
To quantify the glazing properties, three-point bending load-extension curves are shown in Fig. 5b for all the composites. These curves show a practically linear and elastic behavior until failure. Indeed, the combination of the viscoelastic properties of the polymer film and the fragile nature of glass, gives laminated glazing new mechanical characteristics. With the exception of the composite C4, composites reinforced with modified NFC (C2 and C3) show an improvement in extension over PVB. Overall, the C2 composite (40% NFC) represents the highest increases in maximum load with an average of 3907 N while PVB reaches only an average of 3430 N (14% increase).

Young's modulus and extension values increase as the amount of NFC incorporation increases up to 50% (C3), but further addition of NFC (70%) shows a slight decrease in the mechanical properties of the laminated glazing. However, even if the maximum load value is the lowest of all composites, the extension of C4 (3.61 mm) is still close to that of PVB (3.57 mm). By being slightly increased or similar, Young’s modulus values show that the structural properties of our multilayer’s films are similar to a single component film such as PVB. For security glazing, the load and extension are indeed of importance but the absorbed energy at break also. Composites reinforced with modified 40% NFC (C2) and 50% NFC (C3) exhibit the best absorbed energy values with 1.82 J and 1.80 J respectively; while the PVB interlayer absorbs only 1.57 J, (16% and 15% increase). The composite C4 (70% NFC) shows even less performances compared to other composites with a value of the absorbed energy in the order of 1.31 J.

In addition to the good mechanical properties, interlayers composites present an interesting light character. As shown in Fig. 6, for a same area (20 cm^2), the calculated weight of the interlayers in the glazing decreases as the amount of NFC increases in the composite. Indeed, the addition of NFC causes a weight loss of up to 19% (C4) compared to a PVB interlayer only. Composites C2 and C3 exhibit as well a significant weight loss in the order of 7 and 13% respectively. This property is very interesting especially in applications where lightness must be associated with good mechanical properties such as the transport industry. Indeed, studies have shown that a 10% reduction in vehicle weight contributes to an 8% reduction in carbon dioxide (CO_2) emissions (Joost 2012). Figure 6 also shows that thickness does not exhibit a linear trend with decreasing NFC content. C2, composed of 60%PVB and 40% NFC gives the smallest overall thickness and his 44% less than PVB alone and 17% than the C3 composite. This result is in direct relation to the results of C2 in the three-point bending test and further emphasize that an optimal structure could be achieved. Composite C2 clearly exhibit the best overall performance in static testing.

Fig. 6

Calculated weight and thickness of the intercalary composites in a (10×10 cm^2) glazing



Drop weight test of laminated glazing

The laminated glazing tested in this part are D1 (100% PVB), D2 (40% NFC) and D3 (50% NFC), D standing for bigger size glazing samples (30 × 30 cm^2) equivalent to the best candidates obtained from the three-point bending test. To make a precise comparison between the materials, graphs of the temporal evolution of force and energy are presented in Fig. 7. In addition, the measurable values obtained from this test are summarized in Table 3.

Fig. 7

a Impact force–time responses and **b** absorbed energy-time responses of D1 (100% PVB), D2 (40% NFC) and D3 (50% NFC)

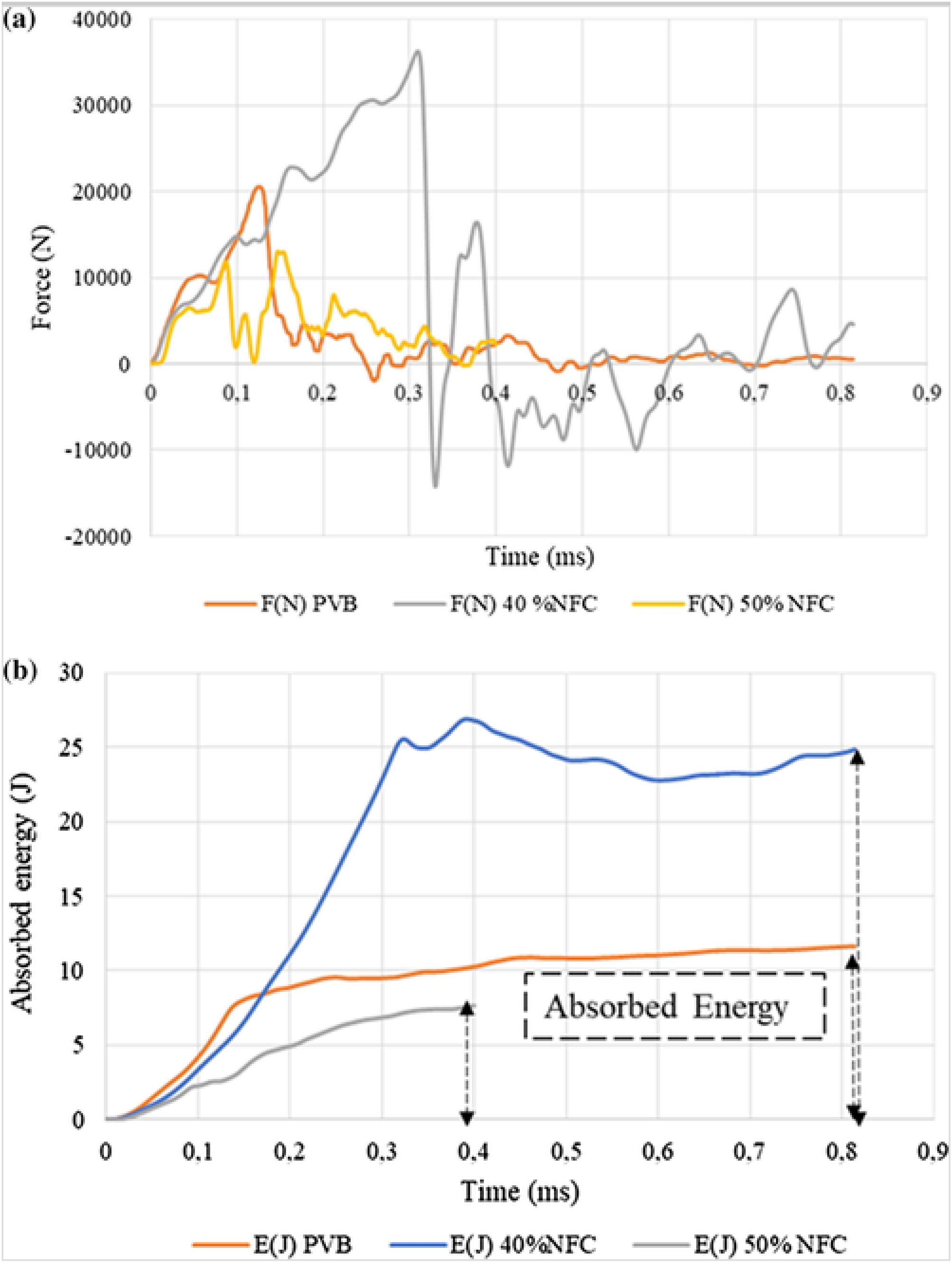


Table 3

Summary of the maximum value of the drop weight testing

	Maximum impact force IF (N)	**Maximum impact energy EI (J)**	**Maximum absorbed energy Ea (J)**	**Ea/EI ratio**	**Response time (ms)**
D1 (0% NFC)	20,529	14.83	14.83	1	0.13

	Maximum impact force IF (N)	**Maximum impact energy EI (J)**	**Maximum absorbed energy Ea (J)**	**Ea/EI ratio**	**Response time (ms)**
D2 (40% NFC)	36,270	26.87	24.81	0.92	0.31
D3 (50% NFC)	11,304	7.63	7.63	1	0.09

Due to the mechanic break needed to get the values on each single D specimen, only one measure is possible. The current measures should then be taken with caution, but seem to tend in the right direction. Indeed, as expected from the static testing, the glazing laminated with composite D2 (40% NFC) still exhibits the best dynamic impact properties. Indeed, the required force to break the D2 glazing is 36,270 N while 100% PVB (D1) reaches only 20,529 N, an increase of 77% (Table 3). As in the static testing, D3 glazing (50% of NFC) shows the lowest properties with a maximum force value not exceeding 12,000 N. However, the value is now about haft of the PVB glazing, which does support the greater importance of a good interfacial adhesion at higher speed. With regard to the impact energy (the maximum value reached, Fig. 7b), the composite reinforced with 40% NFC also shows better results with a value of 26.87 J. It has a gain of energy of 12.04 J compared to interlayer 100% PVB alone (14.83 J, 81% increase). The energy absorbing capacity of glazing laminated with D2 (40% NFC) is quite high (24.81 J) and attractive for many applications, especially in the automotive and construction industries. In our past experiments, the maximum impact energy or the maximum absorbed energy is the governing factor at high impact speed and is of most significance. Another parameter to assess impact resistance is Ea/EI ratio. According to Kannivel et al. (2020), the lower the Ea/EI ratio, the better the impact resistance of the material. In our case, the laminated glazing with D2 has the minimum value Ea/EI of 0.92 (Table 3). For the other samples D1 and D3, the values of Ea and EI are the same (Ea/EI = 1) as shown in Fig. 7b.

The Impact force–time curves of samples (Fig. 7a) show two main peaks when the mass impacts the laminated glazing. The first common peak at about 0.05 ms is attributable to the perforation of the first layer of glass. Afterwards, the samples show different aspects depending on the behavior of the intercalary when it deforms on impact. For example, for sample D2 (40% NFC) additional bumps occurs before the maximum was reached. We also notice that sometimes the impact force does not return to zero, this due to the viscoelastic behavior of the interlayers, which creates a delay in the impactor rebound. The delamination onset is detected by a sharp load drop, followed by violent oscillations as reported by Davies and Olsson (2004). This event corresponds to a well-determined response time value. The high value of D2 response time (0.31 ms) is an indication of the slow passage of the impactor due to the correct arrangement of the composite and fiber-matrix compatibility.

In the end, the static testing (Fig. 5b) and the dynamic testing (Fig. 7) are clearly showing that composite 2 (C2, D2) is superior to PVB in many physical properties with only a slight decrease in light transmission.

The optimum composite composition obtained in our work was 40% NFC and 60% PVB. Usually, in a composite material, the rate of reinforcement is limited by several problems which negatively influence their mechanical properties. The most likely being interfacial defects from fibers to fibers or fibers to matrix decohesion or incompatibility. Therefore, if we exceed a given limits, the achieved materials could exhibit properties well below their potential. As we were targeting substitution of most of the PVB, lower percentage of fiber below 40% were not explored. Still, from the trend between 40, 50 and 70%, 40% seem to be an acceptable compromise and was identified as the best composite arrangement and best fiber-polymer matrix compatibility.

Conclusions

1. We successfully synthesized a laminated glazing using transparent and lighter biocomposite interlayers. Flexural and impact strengths of these laminated glazing were evaluated.

2. The interlayers composites reinforced with modified NFC (NFC/PVB) demonstrate high light transmission values (optimal 93%), a consequence of a better polymer (PVB)-NFC compatibility. This property is maintained even when these biocomposites are assembled between two sheets of glass (10 × 10 cm^2) and the light transmittance values of glazing laminated with C2, 40% NFC (80%) and C3, 50% NFC (78%) remain close to that of PVB alone (85%).

3. The addition of cellulose nanofibers reduces the thickness and the weight of the intercalary by up to 44% and 7% respectively for C2 (40% NFC) compared to PVB alone. Static testing (three-point bend) reveals that the glazing laminated with 40% and 50% reinforced composite interlayer have

respectively 7% and 10% higher extension compared to interlayer of PVB alone. Glazing laminated with 40% NFC/60%PVB presents the highest load value (3907 N).

4. Under dynamic testing (drop weight), candidates C2 (40% NFC) and C3 (50% NFC) were then measured with our control (100% PVB) under an impact test of 226 J. The results obtained with the laminated glazing with biocomposite interlayer reinforced with 40% of NFC are very promising with the highest values in terms of strength and energy. The absorption energy capability and the impact force have increased 67% and 77% respectively compared to PVB interlayer, which made it potentially applicable as an interlayer in security glazing.

5. Further research are underway to confirm the mechanical properties and study optical ones.

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Author contributions

ML: Methodology, Validation, Investigation, Data curation, Formal analysis, Writing—original draft, Visualization. FC: Conceptualization, Cosupervision, Writing—review & editing. EL: Conceptualization, Project administration, Supervision, Writing—review & editing, Funding acquisition.

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Data availability

Not applicable for that section.

Code availability

Not applicable for that section.

Compliance with ethical standards

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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